

DUAL-CANTED UNDULATORS AT THE GM/CA-CAT INSERTION DEVICE BEAMLINES

Requests for beam time at synchrotron radiation beamlines dedicated to macromolecular crystallography have increased significantly in the past several years, and it is anticipated that this trend will continue. In response to this growing demand, the General Medicine and Cancer Institutes Collaborative Access Team (GM/CA-CAT) was established by two partnering institutes at the National Institutes of Health (NIH). The National Institute of General Medical Sciences (NIGMS) and the National Cancer Institute (NCI) are funding the construction and operation of a national user facility for crystallographic structure determination of biological macromolecules by x-ray diffraction at sector 23 of the APS.

Beam time will be available to users through the APS general user program and to grantees of the partnering institutes for special programs. The special program of the NIGMS is the Protein Structure Initiative in structural genomics was formed to study protein structural families and structural folds. This project will require the determination of a large number of protein structures in a high-throughput mode. The NCI has a special program for targeted drug design with the goal of transforming the process by which cancer therapeutics and preventatives are discovered, developed, and tested. Structural analyses will be necessary to determine how candidate drugs interact with their protein targets and to further refine drug design. In common with the structural genomics initiative, this will require the most advanced methods for rapid data collection and structure determination of the widest range of protein types at the highest available resolution.

In order to maximize the x-ray beam available to GM/CA-CAT users, the insertion device (ID) beamline at sector 23 has been equipped with the canted undulator configuration developed by the APS in response to the need for efficient use of APS sectors dedicated to macromolecular crystallography (see "Dual-canted Undulators" in the section, "The APS Light Source"). In the dual-canted undulator geometry [1], two hard x-ray insertion devices are placed co-linearly in the same straight section. The first successful implementation of the dual-canted undulator design was demonstrated in August 2003 at GM/CA-CAT sector 23.

Two insertion device beamlines in one sector can potentially double scientific throughput. But the design of the beamlines should allow them to operate independently while not compromising the performance characteristics of the two lines. The 1-mrad separation between the two beams will not accommodate a modern macromolecular crystallography experimental setup with a large-format mosaic charge-coupled device (CCD) detector. So the x-ray optical design must significantly increase the separation between the two beamlines.

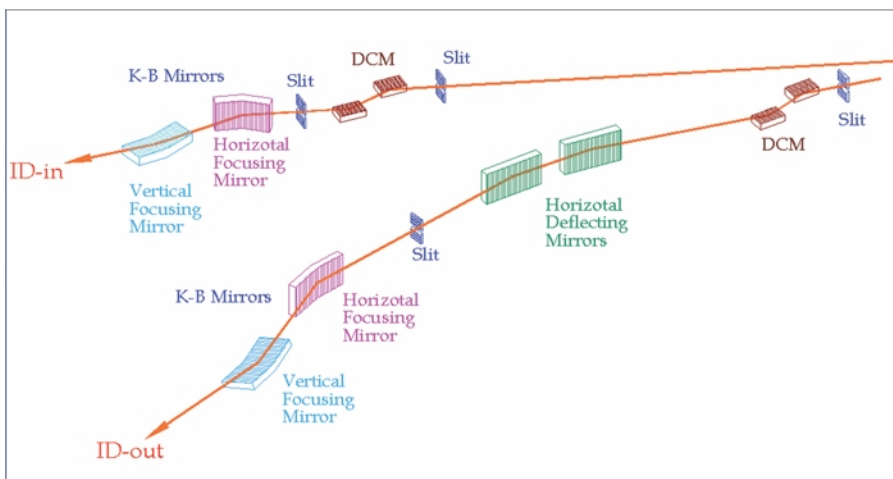


Fig. 1. Schematic view of the x-ray optics of the two ID beamlines.

In seeking a solution to this issue, GM/CA-CAT considered several possible designs, such as large-offset, double-crystal monochromators (DCMs) diffracting in the vertical or horizontal plane; pairs of white-beam mirrors; and pairs of vertical-deflecting monochromatic mirrors. Each of these designs was rejected for various reasons.

One design goal was minimizing the differences between the two beamlines for operational simplicity (Fig. 1). Each beamline employs a DCM to select the x-ray energy. Both the first and second crystals are indirectly cooled with liquid nitrogen, thereby removing potentially leaky cryogenic seals with the silicon. The outgoing monochromatic beam is maintained parallel to the incident white beam because the two crystals are at nearly the same temperature (77K), i.e., they have the same d-spacing. Dispersion of the Bragg angles of the first and second crystals as a function of energy (tuning error) is minimized by using a 160-mm-long second crystal and eliminating a Bragg parallel motion. The beam can be focused in both the vertical and horizontal plane with a pair of mirrors oriented in a Kirkpatrick-Baez (K-B) geometry [2]. These mirrors are constructed of modular piezoelectric bimorph materials [3]. The vertical focusing mirror has four segments, and each segment contains four electrodes. The horizontal focusing mirror has seven segments, each with two electrodes. The bimorph mirrors are shaped to focus the x-rays by applying a voltage to all the electrodes. By applying slightly different voltages to the various electrodes, one can control the detailed shape and minimize the slope error of the mirrors, resulting in a finely focused beam with minimal tails.

The major difference between the two ID beamlines is the location of the DCM and the addition of horizontal deflecting mirrors (HDMs) for the IDout beamline (Fig. 2). The DCM for IDout is positioned further upstream than the IDin DCM (29 m versus 62 m). The white IDin beam and the monochromatic IDout beam are separated by 30 mm in the vertical direction and 30 mm

in the horizontal direction at 30 m from the center of the two undulators. This allows the pair of HDMs to intercept the monochromatic beam sequentially and further deflect the beam horizontally. The mirrors have a unique “T”-shaped cross section to allow clearance for the neighboring white beam. The HDMs will be oriented at a fixed 4.0-mrad angle of incidence, thereby increasing the separation to 17.0 milliradians between the two beams. The resulting horizontal separation in the outboard experimental station, at approximately 56 m from the source, is ~500 mm. The performance characteristics of the two ID beamlines and the bending magnet beamline are summarized in Table 1. The energy of IDout is limited to 20 keV because of the high energy cut-off of the horizontal deflecting mirrors.

The end stations will be designed for streamlined, efficient throughput for a variety of crystal sizes and qualities, representing the cutting edge of structural biology research (Fig. 2). Each ID beamline will be independently tunable for multiwave anomalous dispersion experiments over a wide energy range. They will feature a variable sample-to-source distance, to enhance the control of beam convergence, and rapid-readout, large-format, multi-element CCD detectors, with achievable resolution up to 0.6 Å. Automation of sample mounting, beam alignment, and data collection is incorporated in the design. ○

This work supported by the NIH National Institute of General Medical Sciences, and the National Cancer Institute. ACCEL Instruments, GmbH, Bergisch Gladbach, Germany has been responsible for the detailed design and fabrication of the beamline components. Use of the Advanced Photon Source was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-Eng-38.

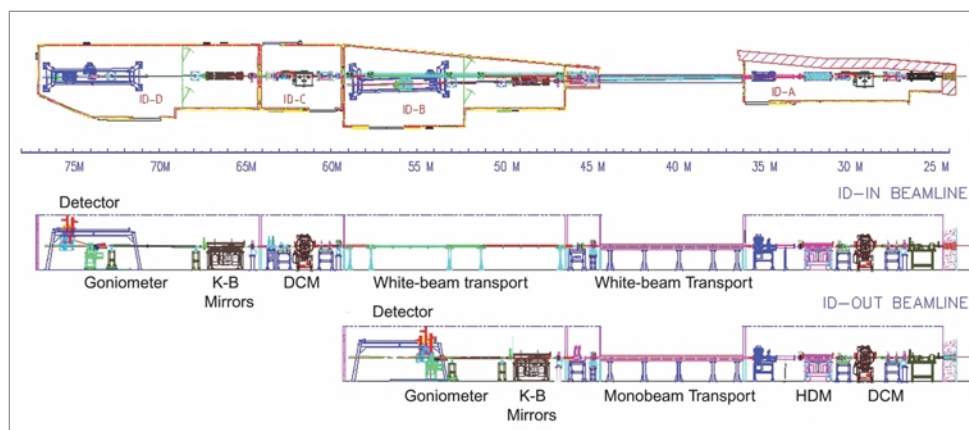


Fig. 2. Plan and elevation views of the two ID beamlines.

See: R.F. Fischetti¹, J.L. Smith², L.E. Berman³, W. Diete⁴, R. Signorato⁴, R. Benn¹, S. Stepanov¹, R. Sanishvili¹, S. Xu¹, A. Urakhchin¹, O. Makarov¹, and W.W. Smith¹, “Insertion Device Beamlines Based on the Dual-Canted Undulator Concept,” in preparation.

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Table 1. ID and BM beamline characteristics.

	IDin and IDout	BM
Energy range (keV)	3.5–35, 3.5–20	3.5–35
Energy resolution (%, full vertical opening angle)	<0.02	<0.02
Flux @ 12 keV (photons/sec/100 mA/0.02% BW)	$>1.0 \times 10^{13}$	$>1.0 \times 10^{11}$
Harmonic contamination (%)	<0.01	<0.01
Rate of energy change at 6.5 keV (eV/sec)	350	350
at 12.0 keV (eV/sec)	3500	3500
Beam position stability after 100-eV change (% of beam size (FWHM))	<5	<5
Beam position stability after 1000-eV change (% of beam size (FWHM))	<10	<10
Beam size at crystal (μm)	50×200 (V \times H)	100×200 (V \times H)
Beam divergence at crystal (mrad)	0.05×0.25 (V \times H)	0.25×2.0 (V \times H)

AN UNPRECEDENTED LOOK AT RIPPLED SURFACES AND INTERFACES OF LIQUIDS

Viewed with the naked eye, the mirror-like surface of a still liquid appears flat beyond measure. Yet, at atomic length scales, such a surface may be as wrinkled as the back of Methuselah's hand. Researchers rely on x-ray scattering to detect and measure such submicroscopic roughness. A team from the University of Chicago (UC) and the University of Illinois at Chicago (UIC) has developed a spectrometer that provides unprecedented spatial resolution of the features on liquid surfaces and liquid-liquid interfaces. With colleagues from Harvard University, and the University of Queensland, Australia, the researchers recently demonstrated the device's potential in a first series of measurements at the APS.

The liquid surface/interface spectrometer at the ChemMatCARS-CAT beamline 15-ID (Fig. 1) takes advantage of the high brilliance and wide energy range provided by the beamline. The beam scatters off a single-bounce steering crystal, which directs the x-rays down on to the sample. After scattering from the liquid surface or interface, the x-rays pass into an x-ray detector. Thanks to the beamline optics, the spectrometer can reach higher energies and momentum transfers than similar devices elsewhere. And that means the spectrometer can resolve smaller spatial features and handle a wider range of samples than rival devices.

For example, the researchers measured the specular reflectivity of the interface between a hydrocarbon known as 2-heptanone and underlying water (Fig. 2). Agitated by thermal motion and tugged by surface tension, the interface should ripple with capillary waves, which affect its reflectivity. According to theoretical calculations, those waves should have wavelengths of about 7 Å. By tracking how the reflectivity varied with the angle between the incident beam and the interface, the researchers confirmed the prediction. The beamline can pump out x-rays with energies up to 32 keV, which can penetrate an unusually wide variety of liquids, including water.

The spectrometer's ability to reach high momentum transfer also makes it ideal for studying atomic-scale layering in liquids, such as liquid tin. Theoretical models suggest that, as with other liquid metals, the surface of liquid tin should have a layered structure, with the electron density varying from layer to layer. The structure should affect how the reflectivity of the surface varies with the angle of incidence. Measurements with the spectrometer confirmed these predictions.

The spectrometer can also accommodate bulky samples, such as large vacuum chambers to study liquid metals or Langmuir troughs for the study of single layers of molecules supported by a liquid's surface. For example, the researchers

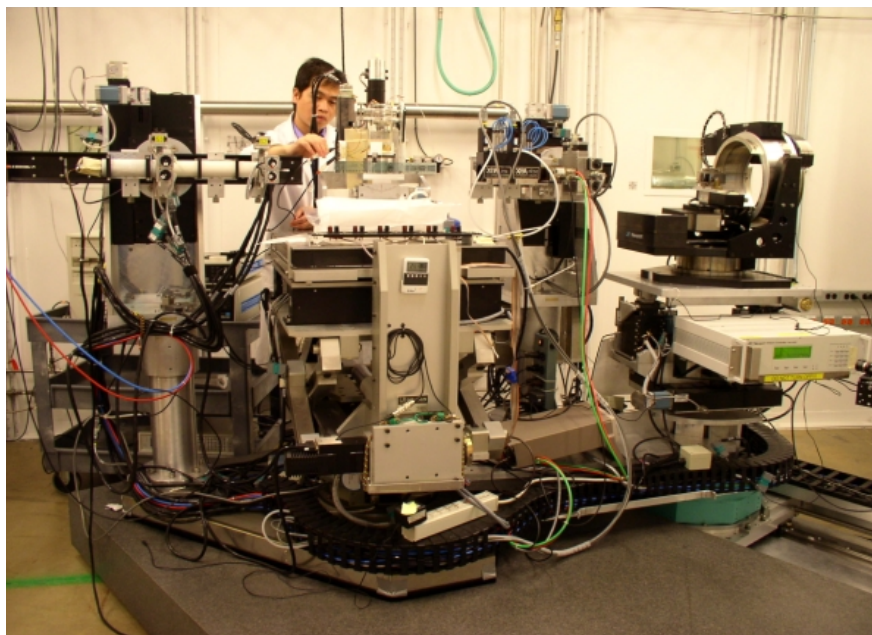


Fig. 1. The liquid surface/interface spectrometer at ChemMatCARS.

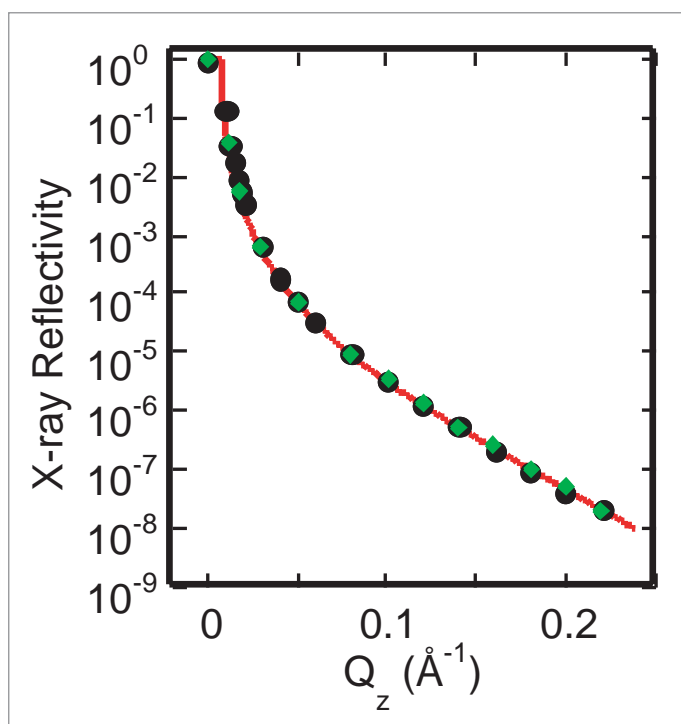


Fig. 2. Data and a fit for x-ray reflectivity from the 2-heptanone/water interface at 25°C. The measured interfacial width is 6.9 ± 0.2 Å and can be compared to the value calculated from capillary wave theory of 7.3 Å. The interfacial width is smaller than the capillary wave value, but the discrepancy is only a 2-sigma effect.

studied the reflectivity of water covered with a monolayer of chain-like surfactant molecules, one end of which attracted water, and the other end of which repelled it. When compared with a theoretical model, the data indicated that the molecules stood out perpendicularly from the surface, like so many blades of grass in a thick lawn. Similarly, the team studied the reflectivity of disk-shaped porphyrin molecules on the surface of salt solutions. In this case, the data suggested that when the film was gently squeezed laterally with a specialized bar, the molecules began to tilt upward like snake scales, instead of lying flat like lily pads.

The researchers also studied the in-plane scattering from the surface of a dilute gallium-thallium alloy. In these measurements, the scattering changed not only the component of the x-ray's momentum perpendicular to the surface, but also the component of the momentum parallel to it. The scattering varies as a function of in-plane momentum transfer, and such variations revealed the structure of the monolayer of thallium on the surface of the alloy. The spectrometer allows experi-

menters to probe higher out-of-plane and in-plane momentum transfers than competing devices.

The first measurements only scratch the surface of the spectrometer's capabilities. The facility is open to general users, and future experiments are sure to add many new wrinkles to the science of liquid surfaces and interfaces. ○

See: B. Lin¹, M. Meron¹, J. Gebhardt¹, T. Graber¹, M.L. Schlossman², and P.J. Viccaro¹, "The liquid surface/interface spectrometer at ChemMatCARS synchrotron facility at the Advanced Photon Source," *Physica B* **336**, 75-80 (2003).

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DEMONSTRATING A FABRY-PÉROT INTERFEROMETER FOR HARD X-RAYS

High-resolution optical devices, such as spectrometers and interferometers, are useful for making precise wavelength measurements in atomic spectroscopy, astrophysics, and many other physical and biological science applications, including use as interference filters and resonators in laser physics. The ability to use such devices when working with x-rays would be highly desirable. However, compared to visible light, x-ray photons have much higher energy and thus smaller wavelengths, making beams of x-rays difficult to reflect off surfaces. If the reflection obstacle could be overcome, x-ray interferometers could be extremely useful as interference filters having micro-eV spectral resolution to study the dynamics of solids, liquids, and macroscopic biological molecules.

Experimentalists from Hamburg University and Argonne National Laboratory, using XOR beamline 3-ID have succeeded in reflecting x-ray light directly backward and producing the first x-ray interference waves by means of a prototype x-ray Fabry-Pérot interferometer. This type of interferometer splits a beam and brings back together portions of the resulting multiple beams to a detector that can measure the interference between them. Since the interfering beams were split from the same initial beam, they were initially in phase. Their relative phase when they meet back at the detector depends on the difference in the lengths of their optical paths. By measuring the phase shifts of the beams, scientists can study the properties of extremely small-scale phenomena.

The primary components of the simplest Fabry-Pérot interferometer are two highly reflecting, low-absorbing parallel mirrors. A standing wave, or multiple reflections, of electromagnetic radiation can be created by adjusting the gap between the mir-

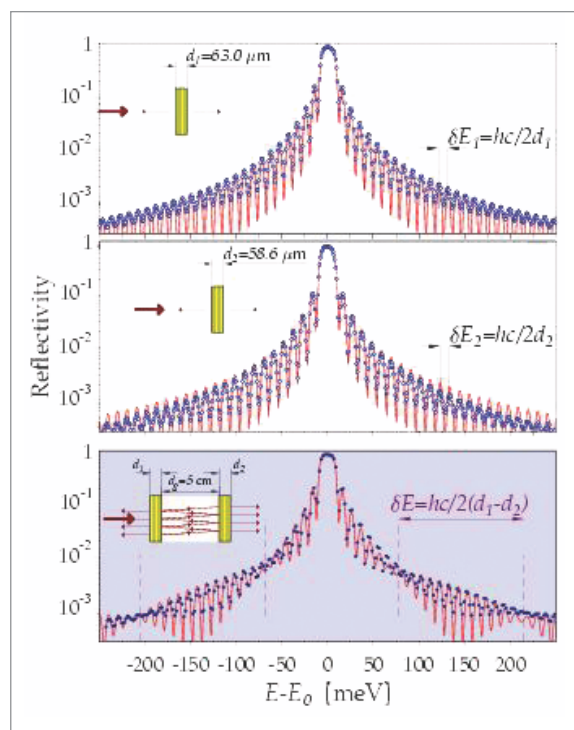


Fig. 1. Energy dependence of reflectivity from the sapphire x-ray mirrors (top) and from the x-ray Fabry-Pérot interferometer (bottom). Additional periodic "beat" modulation in the interferometer spectrum demonstrates a two-crystal interferometric effect (vertical dashed lines). Theoretical spectra (solid red lines) are overlaid on data.

rors to a distance that is one-half the radiation wavelength. Multiple reflections of x-rays had been previously reported in a system with two parallel silicon crystals, but the intensity of the reflected x-ray beams was too low to be useful for interferometry work.

Instead of using silicon crystals, the researchers employed sapphire crystals ($\alpha\text{-Al}_2\text{O}_3$) that provided the necessary high reflectivity. The crystals were grown using a heat-exchange method. Dislocation-free samples were chosen after examining the grown crystals by white beam x-ray topography. The sapphire mirrors were placed at a distance of $d_g \approx 50$ mm and aligned parallel to each other to better than 0.3 microradians. Measurements were performed using x-rays monochromatized to a bandwidth of 2 meV before being directed normal to the mirrors with an accuracy of ± 5 microradians. The researchers measured the energy dependence of the interferometer reflectivity (Fig. 1) and the time dependence of the interferometer response to the excitation with 100-ps pulses of the incident x-ray photons.

As the x-ray energy approached the maximum reflectivity of the crystal mirrors, multiple-reflection signals reappeared every 358 picoseconds, with descending strength observed—the result of the x-rays bouncing back and forth between the mirrors. When the x-ray energy matched the maximum reflectivity, a train of more than 30 peaks was observed, produced by ≈ 60 reflections. This and other experimental data showed direct evidence that the prototype two-crystal x-ray Fabry-Pérot interferometer performed successfully. A set of 0.76 micro-eV broad Fabry-Pérot transmission resonances were measured by the time response of the interferometer. Interference patterns were observed directly in spectral dependences of reflectivity (Fig. 1).

Future efforts will include (1) developing a combined optical/x-ray Fabry-Pérot interferometer (as sapphire is transparent to visible light, and optical sapphire mirrors can be fabricated using thin-film metalization) to directly link the wavelengths of optical photons and Mossbauer photons (in the x-ray part of the spectrum), and (2) developing interference filters with micro-eV spectral resolution for studying the dynamics of solids, liquids, and macroscopic biological molecules. ○

See: Yu.V. Shvyd'ko¹, M. Lerche¹, H.-C.Wille¹, E. Gerdau¹, M. Lucht¹, H.D. Rüter¹, E.E. Alp², and R. Khachatryan², "X-Ray Interferometry with Microelectronvolt Resolution," Phys. Rev. Lett. **90**(1), 013904-1 to 013904-4 (10 January 2003).

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THE HARD X-RAY NANOPROBE BEAMLINE AT APS SECTOR 26

A hard x-ray nanoprobe with a spatial resolution of 30 nm is being constructed at APS sector 26. It uses x-rays with photon energies between 3 and 30 keV. This makes most elements in the periodic system accessible for x-ray fluorescence imaging using K and L atomic transitions at attogram sensitivity. Large penetration lengths (e.g., 140 μm in Si at a photon energy of 10 keV) allow imaging of buried features. Diffraction techniques will be used to probe local strain, structure, and orientation.

Two imaging modes, a scanning-probe mode and a full-field transmission mode, will be combined in a single instrument (Fig. 1). In the scanning probe mode, incident x-rays are focused into a small, diffraction-limited spot on the specimen. Photons from secondary processes, such as fluorescence and scattering, are used for image formation. In the full-field transmission mode, the specimen is illuminated by a condenser system, and a magnified image of the specimen in phase and/or absorption contrast is

recorded by an area detector behind the specimen. In combination, these two modes fully use the radiation properties of an insertion device at a third-generation synchrotron source. In the *scanning probe mode*, the spatially coherent fraction of the undulator beam is used, taking advantage of the high brilliance provided by the APS. The *full-field transmission mode* uses the

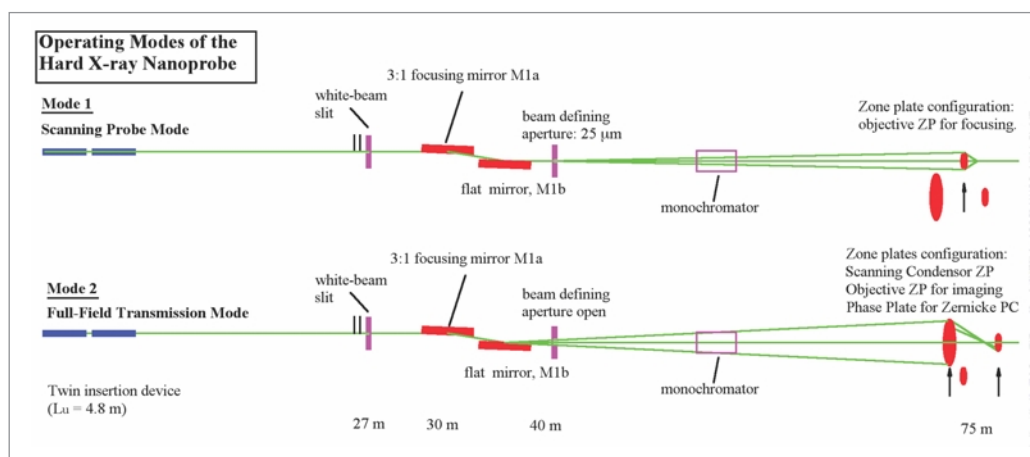


Fig. 1. Design concept for the nanoprobe beamline. An astigmatic beamline design provides spatial filtering in the horizontal direction to match the effective horizontal source size to the vertical beam size. A double-mirror layout provides a fixed exit and higher harmonics rejection. A double-crystal monochromator selects the desired energy. The change between scanning-probe mode and full-field transmission mode is achieved by moving the condenser zone plate, focusing zone plate, and objective zone in and out of the beam.

entire, partially coherent undulator beam. To achieve the highest x-ray optical resolution, we plan to use Fresnel zone plates as focusing and imaging optics.

Two collinear APS undulators A with a period length of $\lambda_u = 3.3$ cm were chosen as the source of high-brilliance x-rays. This will maximize the brilliance and thereby the coherent flux in the existing APS ring layout. To provide spatially coherent illumination for the primary scanning probe mode, the beamline employs spatial filtering in the horizontal direction using a horizontally focusing mirror system at 30 m from the source and a horizontal slit (beam-defining aperture, BDA) at 40 m from the source. The mirror system consists of an elliptically bent, horizontally focusing mirror, M1a, and a plane mirror, M1b. This approach provides a fixed exit angle, improves higher harmonics rejection, and reduces the heat load on the components further downstream in the beamline. Using stripes of Pt, Cr, and Si, this double-mirror scheme allows us to tune the cutoff energy to any value between 3 keV and 30 keV. Because the beam-defining aperture is positioned downstream of the mirror, the mirror is always illuminated along its full length and minimizes thermally induced slope errors. A double-crystal monochromator using Si <111> crystals will be placed 65 m from the source. Since no spatial filtering is provided in the vertical direction, slope errors resulting from heat-induced strain on the first crystal are of concern, and cryogenic cooling will be implemented. Several additional auxiliary beamline components are planned, including a crystal polarizer, a large-bandpass monochromator, and a beam chopper.

The nanoprobe instrument will provide spectromicroscopy, nanospectroscopy, and diffraction contrast imaging as well as full-field imaging of two-dimensional (2-D) and three-dimensional specimens, at a spatial resolution of 30 nm. Critical to achieving these goals are the mechanical stability of the zone

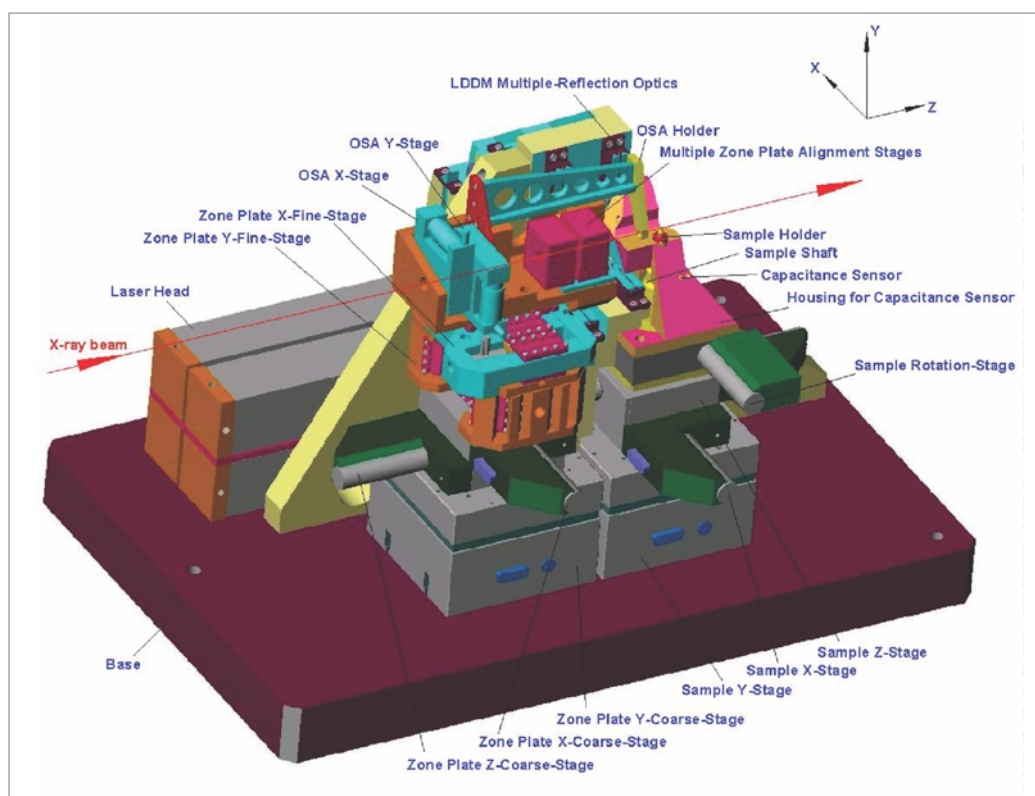


Fig. 2. Mechanical staging and laser Doppler feedback encoder for the hard x-ray nanoprobe prototype instrument.

plate and specimen of 10 nm or better, zone plate motion along the beam axis with a transverse stability of 10 nm, and a single-axis rotation with a run-out of a few 100 nm. High stability is required to allow mapping and imaging at the resolution limit of the zone plates. Motion with high transverse stability is required to achieve nanospectroscopy at the resolution limit. Small run-out of the rotation stage is required to position a specimen for diffraction contrast imaging and to perform tomography in full-field transmission mode.

To provide high-spatial-resolution capabilities to users as rapidly as possible, a prototype instrument that provides these capabilities has been developed (Fig. 2). At the heart of the prototype is a 2-D differential laser Doppler displacement encoder system with a resolution of better than 1 nm. This system monitors the horizontal and vertical positions of the zone plate and specimen stages. The zone plate is mounted on a 2-D flexure stage with a range of 1.5 μ m. Using the output of the encoder system, the flexure allows adjustment for changes in the transverse position of the zone plate and specimen at a rate of 100 Hz or better. The encoder system will be of particular use for nanospectroscopy, where the focal length of the zone plate needs to be changed in step with the incident energy. The flexure stage carrying the zone plate and specimen is mounted on commercial dc-motor-driven translation stages with 0.1-mm resolution and a range of 12 mm. A rotary encoder to provide feedback on run-out during specimen rotation is under development. (Contact G.B. Stephenson, stephenson@anl.gov) ○

USING A DIAMOND ANVIL CELL FOR STRUCTURAL INVESTIGATIONS OF NONCRYSTALLINE MATERIALS AT HIGH PRESSURES

Learning how high pressures affect liquids and other noncrystalline materials at the atomic level would open the door to developing a theoretical understanding of their macroscopic properties—such as their viscosity and self-diffusion, electrical resistivity, compressibility, and thermal expansion—under such conditions. Significant progress became possible in this area with the advent of third-generation synchrotron sources, because their high-brightness x-ray beams enable researchers to use the weak x-ray scattering from these materials in deriving detailed atomic-level structural information about them.

A diamond anvil cell (DAC) is usually used in such experiments when very high pressures are sought. Such a cell essentially consists of a metal gasket placed between two diamond “anvils.” A tiny hole in the gasket serves as a sample container. Pressure exerted on the diamonds forces them together, thus applying a magnified pressure on the gasket and the trapped sample. Diamond is used because it is the hardest material known and is transparent to x-rays and other forms of radiation. Typical DACs, however, are not suited for liquids and amorphous solids, so researchers from The University of Chicago developed and tested a modified DAC designed specifically for these materials.

Diamond anvils are usually mounted on x-ray-absorbing tungsten carbide (WC) seats, which have conical apertures machined into them to provide x-ray access to and from the sample. A larger opening angle would increase coverage of momentum transfer, which is an essential factor for structural studies of noncrystalline materials. On the other hand, the larger opening angle would leave the seats structurally weakened, thus limiting the amount of pressure that can be applied.

To increase the radiation-admitting cone without machining a cone of larger opening angle, the researchers substituted cubic boron nitride (c-BN) for WC as the seat material (Fig. 1). Boron nitride is second only to diamond in hardness and, unlike WC, is transparent to x-rays. To increase the scattered x-ray signal even more, the researchers modified the gasket so that it would accept thicker samples. The modification involved drilling a relatively large hole in the gasket and filling it with boron epoxy. A much smaller hole was drilled at the center of the boron epoxy for sample loading.

This use of boron epoxy also greatly aided data analysis. Subjecting a DAC to an x-ray beam yields a strong x-ray signal from the DAC's components, in addition to the relatively weak signal from the sample. To isolate the sample's scattered x-rays, it is necessary to subtract the DAC's contribution. This is done by repeating the experiment without a sample in place.

For the signal from the empty DAC to serve as a background reference, all parts of the DAC must be identical to

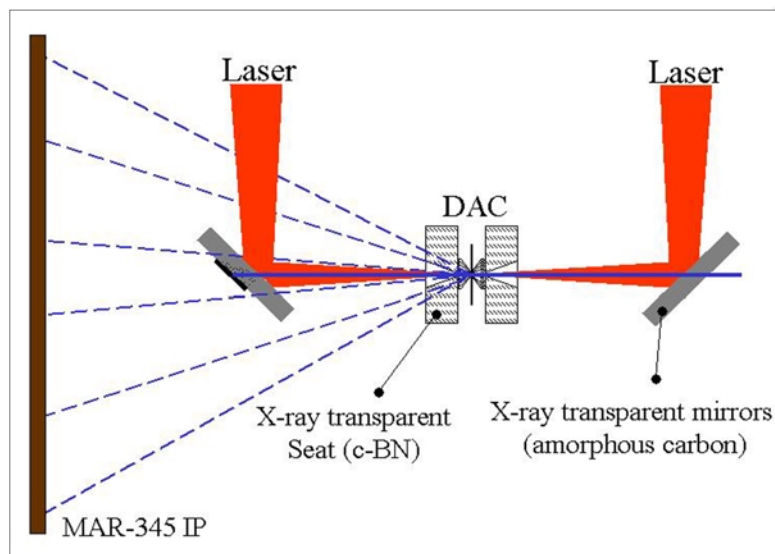


Fig. 1. Schematic of the experimental setup. Samples in the diamond anvil cell can be heated from both sides by a Nd:YLF laser. The laser heating and the x-ray diffraction/scattering and temperature measurements can be made simultaneously with the use of x-ray-transparent, carbon-based mirrors.

those used for high-pressure data collection. Among these parts, the metallic gasket is most critical. Since the metallic gasket hole serves as an aperture for x-ray scattering from the first diamond, great care must be taken to preserve the location, shape, and size of this aperture. Without the boron epoxy, the metallic gasket hole would inevitably deform under high pressures, making it impossible to have an identical aperture for a proper reference. By introducing amorphous boron (or another x-ray-transparent material) in the gasket, the large metallic hole outside the culet area is only weakly affected by high pressures, and deformations occur mainly in the transparent boron-filled area. This results in a metallic aperture close to that used in high-pressure data collection, thus providing a good background reference.

The utility of the modified DAC was demonstrated by using data for amorphous iron collected at 67 GPa at the GSECARS 13-BM beamline at the APS. The DAC's opening angle of 60° and the APS beamline energy of 37.44 keV ($\lambda = 0.3311 \text{ \AA}$) yielded a maximum momentum transfer of 98 nm^{-1} . By further tilting the DAC, the momentum transfer that can be covered was found to increase to 150 nm^{-1} . Researchers learned that the basic polyhedra forming the structure of amorphous iron remain unchanged with pressure.

Although the modified DAC was shown to be ideal for studying amorphous solids, it should be applicable to structural studies of liquids and crystalline materials as well. The large x-

ray access opening should benefit all DAC-based x-ray diffraction measurements, particularly those involving the angle dispersive technique, which is now widely used. The thicker sample capability should aid the study of low-Z materials (low x-ray scattering), while the combination of both of features promises to permit researchers to explore the properties of materials at ultrahigh pressures (>300 GPa), which is important to planetary science. ○

See: G. Shen, V.B. Prakapenka, M.L. Rivers, and S.R. Sutton, "Structural investigation of amorphous materials at high pressures using the diamond anvil cell," *Rev. Sci. Instrum.* **74**, (6), 3021-3026 (June 2003).

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HIGH-TEMPERATURE TRIAXIAL DEFORMATIONS AT PRESSURES TO 15 GPa

To date, knowledge of the physical and chemical processes taking place deep inside the Earth has come primarily from global-scale observations. Earth scientists have recognized, however, that gaining a full understanding of the structural and dynamic properties of the Earth's interior requires the integration of global-scale seismological and other geophysical observations with computer modeling based on laboratory simulations of high-pressure, high-temperature environments. Because of technical difficulties, the effects of high pressure on materials remain far less well studied than those of high temperature or changes in material composition. This situation is changing, however, thanks to new analytical techniques.

Researchers from The University of Chicago, Lawrence Livermore National Laboratory, the University of Colorado, and the State University of New York at Stony Brook collaborated in developing and testing a new high-pressure deformation apparatus that is capable of generating pressures up to 15 GPa at high temperatures (up to ~2000°C). This apparatus is a modification of the cubic-anvil apparatus known as the DIA, which is widely used in solid-media hydrostatic compression experiments that involve synchrotron x-ray diffraction. The apparatus takes advantage of recent developments in the use of synchrotron radiation, thus making it possible to perform quantitative deformation experiments under pressure and temperature conditions that extend down to the transition zone between the Earth's upper and lower mantle.

A DIA consists of six anvils. Four of the anvils face each other in the horizontal plane, while the other two face each other vertically. The square fronts of the anvils thus define a cubic vol-

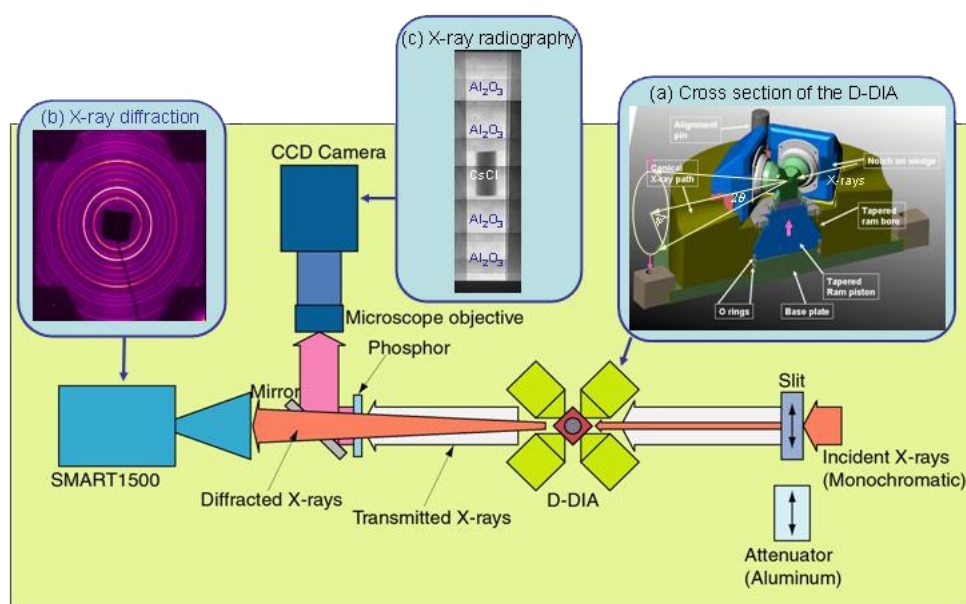


Fig. 1. Current GSECARS experimental setup for high-pressure, high-temperature deformation experiments. (a) The D-DIA, (b) Debye rings used in lattice strain analysis, and (c) x-ray absorption contrast image of sample converted to visible light by using a YAG single-crystal phosphor.

ume at the center of the apparatus. The force of a single hydraulic actuator (or ram) applied along the vertical axis closes the DIA's guide blocks, thus compressing the cubic sample/furnace assembly.

The new apparatus, called the deformation-DIA (or D-DIA), features independent control of the displacements of one anvil pair, which is provided by two additional hydraulic actuators, called differential rams. The differential rams are located within the guide blocks of the D-DIA module and react against the platens driven by the main hydraulic ram that is responsible for hydrostatic pressurization. The unique capability of independently controlling the differential rams allows the imposition of a differential strain and stress field without simultaneously increasing the confining pressure. This capability overcomes a longstanding limitation of multi-anvil devices.

The researchers have successfully tested the D-DIA at both the National Synchrotron Light Source, using the SAM85 multi-anvil system, and at the APS, using the 250-ton hydraulic press at the GSECARS sector 13 bending magnet beamline. Tests with synchrotron x-rays have demonstrated that the D-DIA is capable of generating up to 30% axial strain on 1-2-mm-long samples under confining pressures up to 15 GPa at simultaneous high temperatures. Various compressive strain rates from 10^{-3} to $\sim 2 \times 10^{-6} \text{ s}^{-1}$ have been achieved. Extensional experiments have also been successful. Strains are measured by using x-ray imaging, which has an ultimate length measurement precision of $\sim 0.1 \mu\text{m}$, while pressures are monitored by using standard materials with well established equations of state. X-ray-transparent anvils made of sintered polycrystalline cubic boron nitride have been successfully tested, by using a two-dimensional x-ray charge coupled device detector. Distortions in diffraction lines due to differential stress can be measured with a precision of about 20 MPa.

The D-DIA is open for general users at GSECARS (see Fig. 1), where several high-pressure deformation experiments have been performed. As an example, Fig. 2 shows lattice strains in CsCl (a body-centered-cubic material) determined from 11 reflections during both compression and extension, obtained by researchers from The University of Chicago. Saturation in lattice strain with plastic deformation is highly dependent on Miller indices, indicating strong yield anisotropy, even though the material is nearly isotropic elastically. ○

See: Y. Wang¹, W.B. Durham², I.C. Getting³, D.J. Weidner⁴, "The deformation-DIA: A new apparatus for high temperature tri-axial deformation to pressures up to 15 Gpa," *Rev. Sci. Instrum.* **74** (6), 3002-3011 (June 2003).

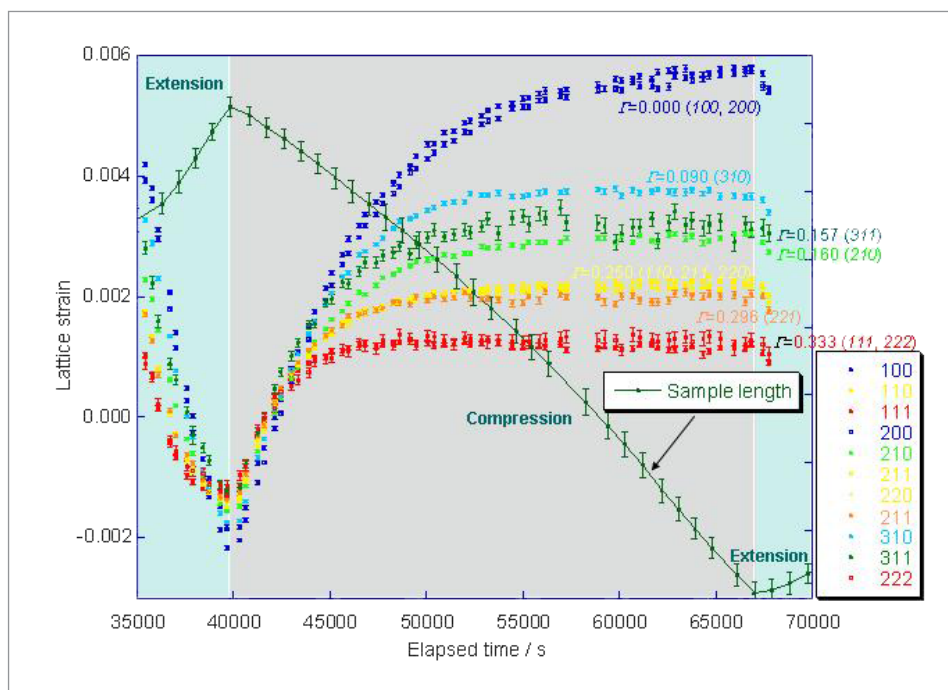


Fig. 2. Measurements of lattice strain in CsCl using 11 diffraction lines, plotted as a function of elapsed time during the first extension-compression deformation cycle. The x-ray wavelength was 0.19 \AA (65 keV); spatial resolution in the length measurements was $1\text{-}2 \mu\text{m}$. These lattice strains can be used to assess differential stress in the sample, through elasto-plastic modeling. Hysteresis loops have been measured by repeating the extension-compression cycles.

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